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(54) **Powdered paint.**

(57) A powdered paint is composed of a particulate group in which the mean particle diameter is in the range of 5 to 20 μm , and the proportion of particles 5 μm and less in diameter is not more than 25 percent by weight.

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BACKGROUND OF THE INVENTION

Field of the Invention

5 The present invention relates to paints, and in particular to powdered paints.

Description of Related Art

10 A general powdered paint is composed mainly of a particle mass produced by grinding pellets obtained from a melted and kneaded raw material containing a binder resin, a pigment, a curing agent, a surface conditioner and the like. The mean particle diameter of the particle mass composing such a powdered paint is usually about 30 μm .

The formation of a film coating of the powdered paint is performed by applying it to an object to be coated through such a method as electrostatic spray coating or fluidized-bed dip coating. The powdered paint is then subjected to a baking treatment, whereby the binder resin in the particle mass melts, forming a continuous film coating.

However, the aforementioned conventional powdered paint cannot provide a large-scale and smooth film coating. In particular, wherein a thin film coating is formed, its smoothness and appearance are liable to deteriorate.

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SUMMARY OF THE INVENTION

An object of the present invention is to form a film coating having good smoothness and appearance.

25 A powdered paint in accordance with one aspect of the present invention is composed of a particle mass in which the mean particle diameter is in the range of 5 to 20 μm , and the proportion of particles 5 μm and less in diameter is up to 25 percent by weight. The powdered paint may further contain fine resin particulates having a glass transition point of 50 to 150 °C, for example.

30 A powdered paint in accordance with another aspect of the present invention can be manufactured by the process comprising the steps of: producing pellets from a melted and kneaded particle mass of raw material for powdered paint, and grinding the pellets into a particle mass in which the mean particle diameter is in the range of 5 to 20 μm and the proportion of particles of diameter 5 μm and less is up to 25 percent by weight.

35 A powdered paint in accordance with a further aspect of the present invention can be manufactured by the process comprising the steps of: producing pellets from a melted and kneaded particle mass of raw material for powdered paint, in which the proportion of the raw material particles of diameter under 700 μm is at least 50 percent by weight; and grinding the pellets into a particle mass.

40 A method of manufacturing a powdered paint in accordance with a still further aspect of the present invention comprises the steps of: producing pellets from a melted and kneaded particle mass of raw material for powdered paint; and grinding the pellets into a particle mass in which the mean particle diameter is 5 to 20 μm and the proportion of particles 5 μm and less in diameter is up to 25 percent by weight. The present method may further comprise a step of mixing the particle mass with fine resin particulates having a glass transition point of 50 to 150 °C, and a mean particle diameter less than that of the particle mass.

45 A method of applying a powdered paint in accordance with a still further aspect of the present invention comprises the steps of: applying a powdered paint to an object to be coated, which paint is composed of a particle mass in which the mean particle diameter is 5 to 20 μm and the proportion of particles 5 μm and less in diameter is up to 25 percent by weight; and performing a baking treatment of the powdered paint.

These and other objects of the present invention will become apparent from the following detailed description.

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DETAILED DESCRIPTION OF THE INVENTION

55 A main component of a powdered paint in accordance with the present invention is a pulverized synthetic resin serving as a binder. This resin may be selected from among thermosetting resins and thermoplastic resins. Wherein a thermosetting resin is used for the powdered paint, the synthetic resin can be epoxy resin, acrylic resin, polyester resin and the like, for example. With an epoxy resin, phthalic anhydride, dicyandiamide, acrylic resins and the like may be added thereto, if desired. With an acrylic resin, polyhydric carboxylic acids, epoxy resins, melamine resins and the like may be added thereto, if desired.

With a polyester resin, polybasic acid, melamine resins, block isocyanate and the like may be added thereto, if desired.

On the other hand, wherein a thermoplastic resin is used for the powdered paint, the synthetic resin can be a vinyl resin such as poly vinyl chloride, polyethylene resin, polyamide resin and the like, for example.

5 The aforementioned powdered paint may contain a pigment such as titanium dioxide, red iron oxide, yellow iron oxide, carbon black, copper phthalocyanine blue, copper phthalocyanine green, and quinacridone red pigment. The powdered paint also may contain a surface conditioner such as polysiloxane or acrylic resin; plasticizers; ultraviolet absorbers; antioxidants; pinhole controlling agent; pigment dispersing agents; and curing catalysts such as amine compounds, imidazole compounds and polymerization
10 initiators; as well as other additives, such as further types of resin. These pigments and additives may be contained in the synthetic resin particles, or may be included as particles other than the synthetic resin particles.

The mean particle diameter of the powdered paint in accordance with the present invention is in the range of 5 to 20 μm , preferably 8 to 16 μm . When the mean particle diameter is less than 5 μm , the fluidity
15 of the powdered paint in heat treatment is lowered so that it becomes difficult to form a dense and uniform film coating. Conversely, when the mean particle diameter is more than 20 μm , the smoothness of the film coating is detracted, and aspects of its appearance such as its glossiness deteriorate.

The mean particle diameter can be measured by an apparatus for measuring particle diameter utilizing laser scattering, such as that manufactured by the SEISIN Corp. and sold under the trademark "LASTER
20 SIZER PRO-7000".

Fine resin particulates may be joined to the surface of each particle composing the powdered paint in accordance with the present invention. The term "joined" means herein either of two situations, in one of which the fine resin particulates adhere to the surface of the particles composing the powdered paint, and in the other of which the fine resin particulates are slightly embedded in the surface thereof. When the
25 powdered paint is thus joined with fine resin particulates, the particles composing the powdered paint are kept from direct inter-particulate contact, which results in preventing the powdered paint from clotting during storage and in improving fluidity during the baking treatment. Consequently, it becomes possible to utilize synthetic resins having a low glass transformation point (Tg point), e.g. 40°C, as a binder component. When the powdered paint is baked, the fine resin particulates thus melt together with the binder resin in
30 forming a film coating. Therefore, the film coating is not liable to exhibit surface irregularities or other such defects in appearance which might otherwise be caused by the fine resin particulates.

These fine resin particulates are preferably made of the same type of resin as the synthetic resin used for the powdered paint. From a practical viewpoint in manufacturing, vinyl resins, acrylic resins, epoxy resins, polyester resins, melamine resins and the like are those usually employed. In particular, vinyl resins
35 are preferred, both with regard to manufacturing simplicity, and in their large potential for design. It is necessary that the resins composing the fine resin particulates not have a Tg point of less than 50°C. Wherein the Tg point is less than 50°C, there is no noticeable effect from the joining of the fine resin particulates. The upper limit of the Tg point, which is not particularly restricted, is usually 150°C. Tg points greater than 150°C do not further effect the outcome. The preferred range of the Tg point is 70 to 120°C.
40 The mean particle diameter of the aforementioned fine resin particulates is set to be necessarily less than that of the powdered paint. The preferred range of the mean particle diameter is 0.001 to 10 μm , more preferably 0.01 to 5 μm . The quantity of fine resin particulates in addition to the powdered paint is preferably adjusted to 0.05 to 35 percent by weight, more preferably 0.1 to 10 percent by weight, expressed as a solid-weight ratio. When the quantity in addition is less than 0.05 percent by weight, there is
45 no noticeable effect from the addition of the fine resin particulates. Conversely, when it is greater than 35 percent by weight, the appearance of the film coating is worsened.

The aforementioned fine resin particulates can be manufactured by, for example, emulsion polymerization or suspension polymerization. Then, they can be obtained by grinding and classifying the manufactured resins by solution polymerization, bulk polymerization or the like.

50 The following describes a method of manufacturing the above-described powdered paint.

First the above-mentioned synthetic resin and pigment, and such additives as a surface conditioner, a plasticizer, an ultraviolet absorber, an antioxidant, pinhole controlling agent a pigment dispersing agent or a catalyst are prepared, and then are blended homogeneous through a mixer. A conventional mixer such as a Henschel mixer, ball mill, or Banbury mixer may be employed.

55 The resulting blend is melted and kneaded. In this process, the blend is heated in a kneader such as an extruder or a heat roller, so that other solid components can be dispersed homogeneously into the melted synthetic resin. Then, pellets are formed from the resulting mixture. Next, the pellets are ground. In this process, a grinding machine such as an atomizer or a jet mill may be employed. A powdered paint is thus

obtained. The powdered paint is treated by a cyclone classifier to remove fine particles so as to satisfy the conditions as above, i.e., wherein the mean particle diameter is in the range of 5 to 20 μm and the proportion of particles 5 μm and less in diameter is up to 25 percent by weight, resulting in the final powdered paint.

5 In the above-mentioned manufacturing process, the raw material for powdered paint which contains a synthetic resin, a pigment and the other additives, preferably contains particles other than those of the synthetic resin, such that the proportion of particles of diameter under 700 μm is at least 50 percent by weight, preferably 60 percent by weight. Such a raw material for powdered paint provides pellets in which the pigment and additive are homogeneously dispersed into the resin component. Consequently, each raw
10 material component composing the powdered paint is homogeneously contained in the particles obtained by grinding these pellets. The amount of particles composed of exclusively the resin component, the pigment component, or the additive component is thereby reduced. Thus, in the film coating formation process to be described, the powdered paint made of the raw material as specified provides a film coating which is superior to the conventional powdered paint in such aspects of appearance as smoothness, since
15 among other things, a homogeneous hardening reaction of the resin is facilitated.

A method of manufacturing powdered paints is not restricted to the above-described method. For example, a mixture, obtained by blending the raw material in a melted state, may be pulverized by a spray drying process.

Wherein fine resin particulates are to be joined with the powdered paint, the above-described fine resin
20 particulates are added to the obtained powdered paint and then mixed. The mixing of the powdered paint and the fine resin particulates can be performed by a conventional mixer such as super mixer, a Henschel mixer, or a Hybridizer ball mill. Conditions under which the powdered paint and the fine resin particulates are combined can be set as desired by properly selecting a mixer and a mixing mode. For example, wherein the Hybridizer ball mill is used, a powdered paint consisting of a particle mass in which fine resin
25 particulates are superficially embedded in the particles can be obtained.

Powdered paints in accordance with the present invention can be applied to various types of articles for painting such as steel plate, zinc phosphate-treated steel plate, or aluminum plate in automobile construction; or sundry electrical appliances, building materials, and the like, for example. When forming a film coating of the powdered paint on such articles, a conventional method such as the electrostatic spray
30 method or the fluidized dip coating method can be employed to apply the powdered paint. The applied powdered paint is then subjected to a baking treatment, whereby the resin component in the powdered paint is melted, forming a continuous film coating.

The powdered paint, which is manufactured from a raw material for powdered paint composed of a particle mass containing 50 percent by weight raw material particles of diameter under 700 μm , contains a
35 pigment and other additives in an approximately homogeneous blend such that its hardening reaction takes place homogeneously when forming a film coating. Therefore, even wherein the specifications as to mean particle diameter and so on as described above are not satisfied, such a powdered paint is yet capable of forming a film coating which is of superior appearance to the conventional powdered paint.

40 DESCRIPTION OF THE PREFERRED EMBODIMENTS

Preparation of Acrylic Resin

Sixty-three parts xylene was charged into a reactor and then heated to 130°C. Next, a monomer
45 mixture containing 45 parts glycidyl methacrylate, 25 parts styrene, 29.72 parts of methyl methacrylate and 0.28 parts 2-ethyl hexyl methacrylate, and 5.0 parts of t-butyl peroctate was drip-added over a three-hour period in an atmosphere of nitrogen. After a 30-minute interval during which the temperature was maintained, one part t-butyl peroctate was further drip-added over a 30-minute period, following which the temperature was sustained for one hour. Next, the solvent was removed by heating to 130°C under
50 reduced pressure, whereby an acrylic resin was obtained. This preparation exhibited a Tg point of 60°C and a mean molecular weight of 3,500, and was employed as the acrylic resin for powdered paints in the following Examples 1 to 7.

Example 1

55 A particle mass of raw material for powdered paint was obtained by blending the following into a homogeneous mixture: 48.2 parts of the acrylic resin; 12.0 parts 1,10-decanedicarboxylic acid (DDA); 0.29 parts benzoin; 0.10 parts polysiloxane surface modifier YF-391f (manufactured by Toshiba Silicone Co.,

Ltd.); and 2.20 parts bisphenol A-type epoxy resin YD-012 (manufactured by Tohto Kasei); wherein the mean particle diameter was 800 μm and the proportion of particles of diameter of under 700 μm was less than 40 percent by weight. The mixture was then melted and kneaded to form pellets with a Co-kneader PR-46 (manufactured by Bus Co., Swiss).

- 5 The obtained pellets were then ground by atomization. The resulting powder was classified so as to produce an acrylic resin powdered paint in which the mean particle diameter was 12 μm and the proportion of particles 5 μm and less in diameter was adjusted to be no more than 25 parts by weight.

Example 2

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A powdered paint was manufactured similarly to Example 1, except that a raw material particle mass in which the mean particle diameter was 800 μm , and the proportion of particles of diameter under 700 μm was not less than 40 percent by weight, was used in place of the particle mass as specified in Example 1.

Example 3

- 15 A solution containing 87 parts methyl methacrylate, 10 parts styrene, and 3 parts methacrylic acid in mixture was drop-added over a 60-minute interval into a reactor charged with a solvent. Following this, the mixture was stirred at 80 °C for 60 minutes, providing an emulsion having 20 percent non-volatile content and particulate diameter in the range of 0.03 to 0.05 μm . The emulsion was spray-dried, producing an acrylic resin fine powder having a 100 °C Tg point.

The finely powdered acrylic resin was added to the acrylic resin powdered paint obtained in Example 2 and mixed in the dry state for 30 seconds with a Henschel mixer. The ratio in mixture of the acrylic resin fine powder was adjusted to be 1.0 percent by weight of the powdered paint.

- 25 Observation through an electron microscope confirmed that this powdered paint was an ultra-fine pulverized resin paint (hereinafter referred to as "UF pulverized paint") in which the acrylic resin fine powder was adhered to the individual particles of the powdered paint.

Example 4

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The finely powdered acrylic resin obtained in Example 3 was added to the acrylic resin powdered paint obtained in Example 1, and mixed homogeneously under the same conditions as Example 3. The ratio in mixture of the acrylic resin fine powder was adjusted to be 1.0 percent by weight of the powdered paint.

- 35 Observation through an electron microscope confirmed that this powdered paint was a UF pulverized paint similar to that of Example 3.

Example 5

- 40 The acrylic resin powdered paint obtained in Example 2 was classified to produce an acrylic resin powdered paint consisting of a particle mass in which the mean particle diameter was 12 μm and the proportion of particles 5 μm and less in diameter was greater than 25 percent by weight.

Example 6

- 45 The acrylic resin powdered paint obtained in Example 2 was classified to produce an acrylic resin powdered paint of consisting of a particle mass in which the mean particle diameter was 30 μm and the proportion of particles 5 μm and less in diameter was greater than 25 percent by weight.

Example 7

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The acrylic resin fine powder obtained in Example 3 was added to the powdered paint obtained in Example 6, and mixed under the same conditions as Example 3. The ratio in mixture of the acrylic resin fine powder was adjusted to be 1.0 percent by weight of the powdered paint.

- 55 Observation through an electron microscope confirmed that this powdered paint was a UF pulverized paint, likewise as in Example 3.

Example 8

A powdered paint similar to that of Example 1 was obtained using a polyester resin powder in place of the acrylic resin powder.

Example 9

A powdered paint similar to that of Example 1 was obtained using an epoxy resin powder in place of the acrylic resin powder.

Comparative Example 1

The pellets obtained in Example 1 were ground by atomization. The resulting powder was classified to produce an acrylic resin powdered paint consisting of a particle mass in which the mean particle diameter was 12 μm and the proportion of particles 5 μm and less in diameter was greater than 25 percent by weight.

Comparative Example 2

The pellets obtained in Example 1 were ground by atomization. The resulting powder was classified to produce an acrylic resin powdered paint consisting of a particle mass in which the mean particle diameter was 30 μm and the proportion of particles 5 μm and less in diameter was greater than 25 percent by weight.

Evaluation

The fluidity and the appearance of film coatings from the powdered paints obtained in the Examples and Comparative Examples were evaluated. The following describes the evaluation method.

(1) Fluidity

The lay angle of the powdered paint was measured. A smaller lay angle indicates better fluidity.

(2) Appearance of Film Coating

The powdered paint was uniformly applied to a steel plate by the electrostatic coating method and was subjected to a 140°C baking treatment for 20 minutes. The resulting film coating was evaluated visually in terms of smoothness and gloss. The evaluation criteria are as follows:

- ⊙ : Very Good
- : Good
- △ : Slightly Poor
- x : Poor

Table 1 shows the evaluation results.

It will be observed that numerous variations and modifications may be effected without departing from the scope of the invention. The preferred embodiments described above are therefore illustrative and not restrictive, the scope of the invention being indicated by the appended claims and all variations which come within the meaning of the claims are intended to be embraced therein.

Table I

		Mean Particle Diameter of Powdered Paint (μm)	Particle-Size Distribution ¹	Raw Material Particle ²	Ultra-Fine Pulverization	Appearance of Coating Film	Lay Angle	
5	Example	1	1 2	A	B	×	⊙-○	5 5°
10		2	1 2	A	A	×	⊙	5 5°
		3	1 2	A	A	○	⊙	4 6°
		4	1 2	A	B	○	○-△	4 6°
15		5	1 2	B	A	×	○-△	5 5°
		6	3 0	B	A	×	△-×	4 0°
		7	3 0	B	A	○	△-×	3 5°
20	Comparative Example	1	1 2	B	B	×	△	5 5°
25		2	3 0	B	B	×	×	4 0°

¹A: Proportion of particles 5 μm and less in diameter was adjusted to be no more than 25 percent by weight.

B: Greater than 25 percent by weight proportion of particles 5 μm and less in diameter.

²A: Proportion of particles less than 700 μm in diameter at least 40 percent by weight.

B: Proportion of particles less than 700 μm in diameter less than 40 percent by weight.

Claims

1. A powdered paint comprising a particle mass in which a mean particle diameter thereof is in the range of 5 to 20 μm , and a proportion therein of particles 5 μm and less in diameter is not more than 25 percent by weight.
2. A powdered paint in accordance with claim 1, wherein said mean particle diameter is in the range of 8 to 16 μm .
3. A powdered paint in accordance with claim 2, wherein said proportion of particles 5 μm and less in diameter is not more than 16 percent by weight.
4. A powdered paint in accordance with claim 3, wherein said particle mass comprises particles of synthetic resin containing at least one selected from the group consisting of a pigment and an additive.
5. A powdered paint in accordance with claim 4, wherein said pigment is at least one selected from the group consisting of titanium dioxide, red iron oxide, yellow iron oxide, carbon black, copper phthalocyanine blue, copper phthalocyanine green, and quinacridone red pigment.
6. A powdered paint in accordance with claim 5, wherein said additive is at least one selected from the group consisting of a surface conditioner, a plasticizer, an ultraviolet absorber, an antioxidant, a pinhole controlling agent, a pigment dispersing agent, and a curing agent.

7. A powdered paint in accordance with claim 6, wherein said synthetic resin is one selected from the group consisting of thermosetting resins and thermoplastic resins.
8. A powdered paint in accordance with claim 7, wherein said thermosetting resin is one selected from the group consisting of epoxy resins, acrylic resins, and polyester resins.
9. A powdered paint in accordance with claim 7, wherein said thermoplastic resin is one selected from the group consisting of vinyl resins, polyethylene resins, and polyamide resins.
10. A powdered paint in accordance with claim 1, further comprising fine resin particulates with a glass transition point in the range of 50 to 150 °C.
11. A powdered paint in accordance with claim 10, wherein said glass transition point is in the range of 70 to 120 °C.
12. A powdered paint in accordance with claim 10, wherein a mean particle diameter of said fine resin particulates is less than that of said particle mass.
13. A powdered paint in accordance with claim 12, wherein said mean particle diameter of fine resin particulates is in the range of 0.001 to 10 µm.
14. A powdered paint in accordance with claim 13, wherein the proportion of said fine resin particulates is in the range of 0.05 to 35 percent by weight.
15. A powdered paint in accordance with claim 14, wherein said fine resin particulates are particulates of a resin selected from the group consisting of vinyl resins, acrylic resins, epoxy resins, polyester resins, and melamine resins.
16. A powdered paint manufactured by the process comprising the steps of:
melting and kneading a raw material for powdered paint and producing pellets therefrom; and
grinding said pellets into a particle mass in which a mean particle diameter thereof is 5 to 20 µm, and a proportion therein of particles 5 µm and less in diameter is not more than 25 percent by weight;
17. A powdered paint in accordance with claim 16, wherein at least 50 percent by weight of said powdered paint raw material is particles less than 700 µm in diameter.
18. A powdered paint in accordance with claim 17, further comprising fine resin particulates having a glass transition point in the range 50 to 150 °C and a mean particle diameter in the range of 0.001 to 10 µm.
19. A powdered paint manufactured by the process comprising the steps of:
melting and kneading a particle mass of synthetic resin and raw material for powdered paint resin, wherein at least 50 percent by weight is particles of less than 700 µm diameter, and producing pellets therefrom; and
grinding said pellets into a particle mass.
20. A powdered paint in accordance with claim 19, wherein said raw material particles include pigment particles and additive particles.
21. A method of manufacturing a powdered paint comprising the steps of;
melting and kneading a raw material for powdered paint and producing pellets therefrom; and
grinding said pellets into a particle mass in which a mean particle diameter is 5 to 20 µm, and the content of particles 5 µm and less in diameter is not more than 25 percent by weight.
22. A method of manufacturing a powdered paint in accordance with claim 21, wherein said raw material contains a synthetic resin and at least one selected from the group consisting of pigment particles and additive particles.

23. A method of manufacturing a powdered paint in accordance with claim 25, wherein said pigment particles and said additive particles are of less than 700 μm diameter.
- 5 24. A method of manufacturing a powdered paint in accordance with claim 23, wherein said powdered paint raw material contains not less than 50 percent by weight of said pigment particles and said additive particles.
- 10 25. A method of manufacturing a powdered paint in accordance with claim 24, further comprising the step of mixing said particle mass with fine resin particulates having a glass transition point in the range of 50 to 150 °C and a mean particulate diameter of less than that of said particle group.
26. A method of manufacturing a powdered paint in accordance with claim 25, wherein said particle mass and said fine resin particulates are mixed using a hybridizer ball mill.
- 15 27. A raw material comprising a synthetic resin, and additive particles which include pigment particles and of which diameter is less than 700 μm .
28. A raw material in accordance with claim 27, containing not less than 50 percent by weight of said additive particles.
- 20 29. A powdered paint coating application method comprising the steps of:
applying to an object to be coated a powdered paint consisting of a particle mass in which a mean particle diameter thereof is 5 to 20 μm and a proportion therein of particles 5 μm and less in diameter is not more than 25 percent by weight; and
25 treating said powdered paint by a baking process.
30. A powdered paint coating application method in accordance with claim 29, wherein said powdered paint is applied by one of either the electrostatic spray method and the fluidized dip coating method.
- 30 31. A powdered paint coating application method in accordance with claim 30, wherein said object to be coated is one selected from the group consisting of an iron plate, a zinc phosphate-treated iron plate, and an aluminum plate.

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P,X	EP-A-0 497 526 (NIPPON PAINT CO., LTD.) * claims 1,2 * * page 2, line 7 - line 11 * * examples A.1-A.4; table 2 * * Comparative Example A.4 * ---	1-7,16, 21,22, 29,30	
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Place of search THE HAGUE		Date of completion of the search 22 JANUARY 1993	Examiner HOLLENDER C.J.F
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document I : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- A : member of the same patent family, corresponding document			

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CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- @ : member of the same patent family, corresponding document			